Three-Dimensional Visualization of Microstructures

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Abstract

This case study describes a technique for the three-dimensional analysis of the internal microscopic structure ("microstructure") of materials. This technique consists of incrementally polishing through a thin layer (approximately 0.2 μm) of material, chemically etching the polished surface, applying reference marks, and performing optical or scanning electron microscopy on selected areas. The series of images are then processed employing AVS and other visualization software to obtain a 3D reconstruction of the material. We describe how we applied this technique to an alloy steel to study the morphology, connectivity, and distribution of cementite precipitates formed during thermal processing. The results showed microstructural features not previously identified with traditional 2D techniques.

1 Introduction

The influence of the microscopic structures (or "microstructure") on the mechanical properties of materials is well established [3]. For example, when certain thermal processing procedures are applied to steel, plate- or needle-shaped precipitates are formed. Precipitates with these elongated shapes can be deleterious to fatigue strength and fracture toughness in low-carbon steel welds [5]. To fully understand the interaction between structure and properties, it is critical that materials scientists understand the three-dimensional shape of microstructures. This allows control of microstructural evolution, and therefore mechanical properties, during the thermomechanical processing of materials.[1] This requires the application of 3D visualization techniques to the fields of metallurgy and materials science.

In the past, many authors have characterized the morphology and distribution of precipitates by using conventional optical and electron microscopy techniques [4, 1, 6], which typically involve studying 2D slices or planes of polish of the material. However, these traditional methods suffer from a serious drawback: even simple three-dimensional shapes often cannot be deduced from examining random planes of polish, much less the complex morphologies and distributions of grains and precipitates found in many materials [11]. In recognition of these problems, early attempts at 3D visualization combined serial sectioning with motion picture films [7]. Visualizations were thus limited to viewing the sequence of images parallel to the sectioning direction. Only in recent years have serial sectioning techniques been enhanced by computer visualization to obtain more useful 3D information [10]. Although there have been a few previous attempts at applying visualization to materials science research [13], there is very little literature in the field regarding the 3D visualization of microstructures.

This paper reports on the metallurgical application of a serial sectioning and three-dimensional visualization technique developed at the Naval Research Laboratory. We apply three-dimensional reconstruction and visualization to a sequence of images which were collected during the serial sectioning of an alloy steel. This approach is relatively new to the field of metallurgy [12, 10, 8]. This study shows that the 3D nature of microstructures is not easily predicted from looking at isolated 2D slices of the material. The 3D visualization technique described allows metallurgists to understand the 3D distribution and morphology of microstructural features [8], and the results have challenged the conventional thinking about how these features form and evolve.

2 Material Preparation

We studied a sample of high-purity steel, composed of iron alloyed with 1.3 wt. % carbon and 13.4 wt. % manganese. This alloy was processed in the isothermal heat treatment facility at the Naval Research Laboratory. Figure 1 shows a plane of polish of this alloy, which reveals a 2D slice of its microstructure. The light gray areas are the major phase, called austenite, which is the face-centered cubic form of iron. The dark boundaries delineate individual austenite grains. These grains are outlined by a film of the minor phase, called cementite (Fe_3C), which precipitates out along the austenite boundaries during heat treatment. The cementite phase also appears as elongated or blocky precipitates within the austenite grains. Because the cementite phase is more brittle than the austenite, the strength, ductility, and other mechanical properties of the alloy are influenced by the cementite morphology and distribution.

This particular alloy has two properties which facilitated our experiments: 1) there is strong optical contrast between the two phases, and 2) it is possible to selectively remove the austenite phase with deep etching techniques, while leaving the cementite intact. The combination of deep etching and scanning electron microscopy allowed experimental verification of the results we obtained through visualization techniques.

We prepared 250 sections, with an average spacing of 0.17 $\pm$ .07 μm between layers, by incrementally polishing with a Buehler Texmet cloth using an abrasive 0.06 μm silica slurry. We controlled the sectioning depth by calibrating the polishing pressure and time. We then lightly etched the polished surface of each layer with a 4% solution of nitric acid in methanol (nital). We applied fiducial hardness indents using a Buehler Micromet II hardness tester with a Vickers hardness indenter at a 10g load. After each polishing step, we performed optical microscopy at a magnification of 1000X using an oil immersion objective lens, and acquired digital video images using PGT Imagist image analysis software on a Sun SPARCstation 5 computer. We studied four overlapping areas encompassing a total of approximately 150 μm x 180 μm.

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[1] Thermomechanical processing is the mechanical processing (such as rolling, forging, etc.) in conjunction with heat treating (annealing, aging, continuous cooling, etc.) that is used in the manufacturing process of many metals.

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14. ABSTRACT
This case study describes a technique for the three-dimensional analysis of the internal microscopic structure (?microstructure?) of materials. This technique consists of incrementally polishing through a thin layer (approximately 0.2 m) of material, chemically etching the polished surface, applying reference marks, and performing optical or scanning electron microscopy on selected areas. The series of images are then processed employing AVS and other visualization software to obtain a 3D reconstruction of the material. We describe how we applied this technique to an alloy steel to study the morphology, connectivity, and distribution of cementite precipitates formed during thermal processing. The results showed microstructural features not previously identified with traditional 2D techniques.

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3 3D Visualization

The images were registered with respect to each other by aligning the hardness indents using NIH Image version 1.60 and Adobe Photoshop version 3.0 on a Power Macintosh 7200/120 personal computer. The importance of proper alignment through the use of fiducial marks has been demonstrated [11]. We calculated thickness increments from the known indenter diagonal:depth ratio by measuring the change in diagonal length of the hardness indents after each polishing step. We assumed that each successive polishing plane was parallel—the validity of this assumption was demonstrated by the lack of divergence in the diagonal dimensions of several widely-spaced hardness indents as the sectioning process progressed. Additionally, sequential etching showed that the light etch in 4% nital did not significantly affect the microhardness diagonal dimension. Figure 2 shows six representative images through the material, each separated by approximately 2 μm or 10 sections.

The regions of interest, that is, the cementite precipitates, were marked by the material scientist according to his criteria of which features are simple noise and which ones are actual cementite. The ones determined to be noise were manually removed and the ones determined to be cementite precipitates were manually emphasized. This manual alignment and de-noising does not produce large amounts of error or uncertainty, as the overall shape, distribution, and connectivity of the precipitates are more important than the fine-scale details of the shape. This whole process of marking was very tedious work, but it cannot be avoided, as a filtering routine would not recognize the fine-level details of the structures.

Once this process was carried out for all 250 sections of the material, we visualized the stack using AVS [2]. First, we combined the images to form an AVS volume dataset. Then, using various AVS image processing modules to reverse image highlights and reduce random noise in the images, we displayed the resulting 3D dataset using the AVS "tracer" module. The tracer module generates a 2D ray-traced, volume-rendered image (Figure 4). We also generated isosurfaces from the volume dataset, and then visualized them using the AVS geometry viewer (Figure 3).

4 Results

The series of images obtained from the serial sectioning method were viewed both as a video sequence that steps through the material slice-by-slice, and as 3D reconstructions. Both methods reveal important 3D information that 2D techniques are unable to provide. While some 3D information could be inferred from observing the video sequence, the visualization of the 3D reconstructions was superior as it provided the ability to view the entire structure from any perspective.

The computer reconstruction and visualization of serial sections has revealed the true three-dimensional cementite precipitate morphologies, distributions, and interconnectivities. We coupled the ability to rotate the reconstruction to any perspective with the ability to calibrate the length scale of the image to collect the three-dimensional measurements of over 200 precipitates. This allowed us to quantify the shapes through direct measurements of their length, width, and thickness, as well as by observing the precipitate interconnectivities. These are the first measurements of this type in materials science.

The reconstructions in Figures 3–5 reveal the distribution and morphology of the cementite precipitates as follows. The matrix (austenite) grain boundaries are coated with a cementite film; holes in the film confirm that only partial coverage of the austenite grain boundaries is obtained. Elongated lath-shaped cementite precipitates project from the boundary into the grain interiors. All of the lath- and plate-shaped cementite precipitates observed in this investigation make contact at some point with the cementite films covering the austenite grain boundaries. Thus, truly intragranular cementite precipitates have not been observed in 3D, in contradiction to what is suggested by observations from 2D sections.

This three-dimensional analysis revealed several aspects of cementite precipitates that were previously unrecognized, and has enabled the number of classifications in Dube's morphological classification system [4] to be reduced from nine to only two for this alloy. In particular, we found that "grain boundary allotriomorphs" grow primarily within the plane of the grain boundary and exhibit a dendritic form at early stages of growth, and "Widmanstdtten precipitates" extend into the austenite grain, are thin in one dimension, and can vary widely in length-to-width ratio from laths (approximately 15 to 1) to plates (approximately 1 to 1).

These results highlight two problems associated with using two-dimensional images to characterize three-dimensional features: (1) single images from random planes of polish introduce ambiguities that mask the larger-scale three-dimensional shapes and interconnectivities of precipitates, and (2) subtle topographic features of precipitates are not revealed in two-dimensional images from random planes of polish.

We employed a deep-etching process to supplement and corroborate the results of the 3D visualizations [9]. With this method, we selectively removed the austenite phase by chemical etching, leaving the cementite precipitates intact for direct observation by scanning electron microscopy. These studies verified the distribution and morphology of the cementite precipitates.

5 Additional Visualizations

5.1 3D Physical Models

In addition to the computer visualization, we obtained an opportunity to create 3D physical models using the University of Michigan Rapid Prototyping facility. We began by filling in the original images to make the cementite precipitates more solid. We smoothed the resulting 3D dataset using a $3 \times 3 \times 3$ Gaussian kernel, and tri-linearly interpolated from $168 \times 181 \times 155$ to $256 \times 256 \times 128$ in size. Since the prototyping facility required a binary dataset, we thresholded the dataset at a 50% intensity value. We produced both wax and nylon models of $3'' \times 3'' \times 1.5''$ in size. The resulting surface appear very similar to the original rendered images, and verify the visualization results. Figure 5 shows a photograph of the wax model.

5.2 GROTTO Visualization

We also explored visualizing the microstructures in a fully immersive virtual environment, using the GROTTO (Graphical Room for Observation, Tactical Training, and Orientation) device of the Virtual Reality Lab at the Naval Research Laboratory. The GROTTO is a CAVE^{TM}-like system, that is, a stereoscopic surround-screen, surround sound, virtual reality display device. We used a 3D joystick and a head-tracking device to interact with the dataset in the GROTTO. The 3D joystick allows the user to manipulate the dataset, and the head tracking device adjusts the viewing frustum. This allows the user to view all sides of the dataset by walking around it. We developed a virtual panel with several virtual instruments for interacting with scientific datasets, which allow scientists to transform the virtual image in various ways.

For a smooth image, as well for the best possible resolution in the GROTTO, we performed a tri-cubic interpolation of the original AVS 3D dataset, which quadrupled the number of grid points. We used this grid to build a generic AVS module, which we then transformed into VRML (Virtual Reality Modeling Language), which could be read by the GROTTO libraries. When we first created it, we could not interact with the VRML dataset in real-time, because

it contained too many triangles and was too slow to render. We therefore had to limit the dataset to 500,000 triangles.[2] We used triangle decimation techniques to achieve this limit. These routines aim for a target reduction rate while conserving a certain level of detail. In this case, we needed a vertex reduction factor of approximately 10. None of the decimation routines we used were able to deliver this reduction factor; their reduction factors were either too small ($\approx$5) or too large ($\approx$50). We discovered that the best way to optimize the triangle decimation routines while preserving the desired level of detail was to use two or more routines in a nested, hierarchical fashion. In this way, we managed to obtain intermediate reduction rates and were able to reduce the total number of triangles in the model by a factor of 30.

The scientific visualization virtual toolbox allowed scientists to inspect both the austenite grains and the cementite precipitate network as a whole, and then to separate and study individual cementite precipitates. The three-dimensional effect produced and the size of the images displayed provided a compelling sense of immersion. This gave the scientists a real sense of the morphology of the microstructures and the spatial distribution of the precipitates, which cannot be matched by a desktop computer monitor.

During one of the GROTTO sessions an important discovery was made by one of us (G.S.): we often find a hole in the cementite grain boundary film very near the base of cementite precipitates. This may indicate some relationship between precipitates and holes that had not been observed. While these structures can be observed on the desktop, we had generally not noticed them until the examination in the GROTTO. This shows, to some degree, that the immersive display of the GROTTO may make it easier for scientists to observe qualitative visual aspects of three-dimensional information. We also feel the GROTTO shows great potential as a device for the communication and dissemination of this type of visual information.

6 Conclusions and Lessons Learned

The understanding and visualization of the 3D morphology and distribution of grains and precipitates is important for the control of microstructural evolution and thus the mechanical properties of materials. We have presented a combination of experimental and visualization techniques which are relatively new to the field of metallurgy. These methods allow us to study three-dimensional microstructural features that have previously only been studied with two-dimensional techniques. We have discovered several new features of cementite precipitates in high-carbon steel that were previously unknown. Among these findings is the observation that cementite precipitates that appear to be isolated within the austenite grains are actually in contact with the grain boundaries. The visualization techniques described here can be applied to the entire materials field, and future plans include extending these studies to another steel alloy.

The visualization process led to a variety of interesting visualization problems that are currently being investigated in detail. The way the image processing had to be performed visually by a human operator is an area that could possibly be automated by pattern recognition techniques. Of equal importance is the problem of triangle decimation; here we are performing an evaluation of decimation routines and looking for new algorithms to develop a more efficient technique. Interaction with the dataset in the GROTTO could also be improved; we are trying to discover the principles behind simple, natural ways of interacting and manipulating scientific datasets in this virtual environment. *Platform hopping* was another problem for this project, as the images had to be manipulated on four different platforms: a Sun workstation for image acquisition, a Macintosh to perform the de-noising and alignment of the images, an SGI for the AVS visualization, and an Abekas for video production.

This project involved the use of several three-dimensional visualization techniques. The ray-traced image and isosurfaces from AVS gave the general structure of the grain and precipitates, and all all of our results can be obtained from the study and analysis of these visualizations. Nevertheless, the GROTTO display helped us make an interesting qualitative observation which we had not before noticed. On the other hand, it is still relatively cumbersome for the scientist to perform scientific visualization inside the GROTTO. Our dream interaction technique would allow scientists to manipulate their virtual models in the GROTTO with the same degree of simplicity with which they can manipulate a 3D physical model.

[2] The reason for this triangle limit is: in order to render an object in stereo, we have to render both the left- and right-eye images, which doubles the number of triangles being rendered. Then, we have to multiply by the number of screens (4 in the case of the GROTTO). Our hardware (a 6-processor ONYX 2 SGI computer) can render 4 millions triangles while allowing real-time interaction. Thus, the maximum number of triangles that our rendering hardware provides in the GROTTO is about 500,000.

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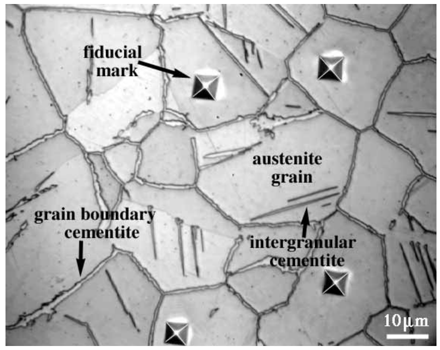

Figure 1: An optical photomicrograph of the Fe-1.3 wt. % C - 13 wt. % Mn alloy.

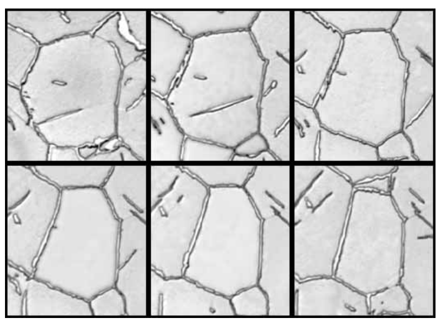

Figure 2: A series of images through the material, every tenth section (2 μm) is shown.

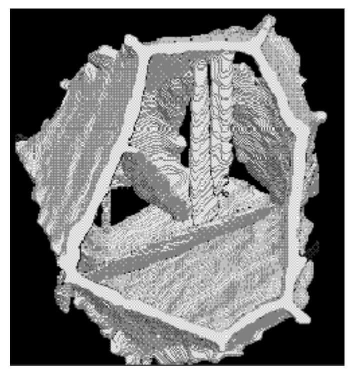

Figure 3: Isosurface.

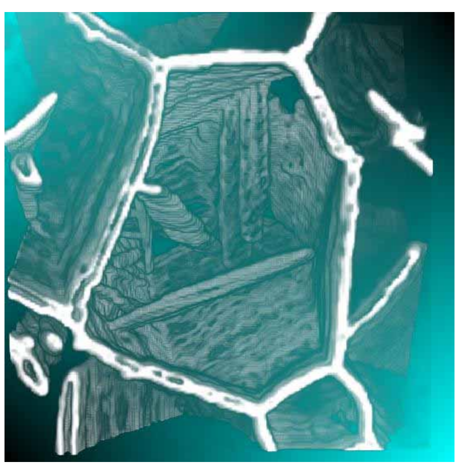

Figure 4: Ray Traced Image.

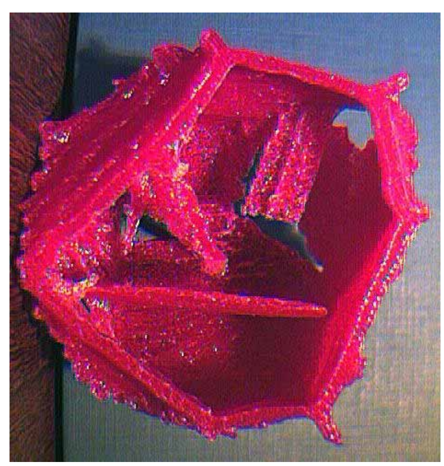

Figure 5: Rapid Prototype Model.